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Experimental Unit for Recovery of Volatile Flavors

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Full-flavor concentrated apple juice, which on the addition of water formed a beverage almost equivalent in taste and aroma to fresh juice, had been made with the aid of a process and apparatus devised by some of the present authors and their former associates. This apparatus stripped the aromatic constituents from the juice and concentrated them into an "essence" for completion of the full-flavor juice concentrate. On applying that apparatus to the juices of other fruits, the need for certain improvements, to obtain a satisfactory concentrate, became evident.

The improved apparatus is described, and diagrams and dimensions are given for a small pilot plant unit. With this apparatus, substantially complete recovery of the volatiles is achieved, as well as pasteurization, and all with much less heat damage to the juice than occurs in conventional pasteurization. Heating and vaporization take only 1 second, and total heat exposure time is adjustable to as little as 2.4 seconds.

This apparatus enables investigation of the effect of heating time on the quality of fruit juice concentrates. Various degrees of stripping and of concentrations of essence and various systems of recovering aromas from the vent gases can be studied. Juices hitherto impossible to process without heat damage can be handled by this ultra-rapid processing.

IN 1944 a process was developed at the Eastern Regional Research Laboratory for recovering the volatile flavors of apple juice in concentrated unaltered form (*4*). The process was later improved (*5*). The volatile flavor concentrate was called apple essence; when it was added to highly concentrated apple juice a full-flavor concentrate was obtained. Since that time the process for essence recovery has been applied to the juices of other fruits and berries (*2*). More recently essence recovery was applied to the preparation of full-flavor fruit juice concentrates—Concord grape concentrate (*3*), frozen concentrate from western apples (*6*), and frozen concentrate from Concord grapes. Currently studies are being made at the Eastern Regional Research Laboratory on frozen apple juice concentrates containing essence and made from eastern varieties. The manufacture and use of volatile fruit flavo concentrates are subject to regulations of the Bureau of Internal Revenue (*1*).

This publication describes a new experimental unit for essence recovery, adaptable to the determination of optimum conditions for the recovery of fruit juice volatiles. The time during which the juice is held at high temperature is adjustable, for some juices are injured by heat and others are improved; with this apparatus the time can be reduced to 2.4 seconds, a time much shorter than previously employed. Even this shortened time, however, adequately pasteurizes the juice

SCHEME OF APPARATUS

When studies on the recovery of volatile flavors from fruit juices were extended to other fruits than apple, it became apparent that some modifications in equipment were required.

In the case of some juices it was found necessary to vaporize more than the 10% which is required to strip the volatile aroma from apple juice. With certain juices the volatile aromas were not entirely recovered by scrubbing the vent gases with chilled essence, though this had been found satisfactory for apple juice. Some juices are more heat-sensitive than apple juice. There was therefore designed an experimental unit for essence recovery, with improvements which would overcome these difficulties as well as others that had been encountered, and at the same time would be capable of pasteurizing the juice without damaging its flavor by heat as does conventional pasteurization. This unit was designed to have a capacity of 10 gallons per hour of juice when vaporizing 10% of the juice, or 5 gallons per hour when vaporizing 20%. It has proved a valuable tool in varying the percentage of vaporization, the flow rate, the strength ("volumetric fold") of the essence, and the heating time to determine the operating conditions best suited for a given juice. Studies of this sort on grape juice and on apple juice are currently under way at the Eastern Regional Research Laboratory, but the data obtained are not yet sufficiently comprehensive to be reported.

The volumetric fold of the essence is defined as the volume of juice processed in obtaining one volume of essence. If the stripping of the aroma from the juice and its recovery in the essence are both 100% efficient, the essence will have an actual strength or potency, with respect to the volatile constituents, expressed by the same figure as the volumetric fold.

Figure 1 shows the arrangement diagrammatically; Figure 2 is a photograph of the unit. The fundamental principle of operation, as in the previous units developed at the Eastern Regional Research Laboratory, comprises rapidly vaporizing a small portion of the juice to strip the aroma from it, fractionating the vapor to concentrate the aroma into an aqueous essence, and scrubbing by chilled liquid the vent gases from the condenser of the fractionation column.

In the present apparatus the juice is pumped from feed tank *A* (Figure 1) through preheater *B* and vaporizer *C* to separator *D*, whence emerge stripped pasteurized juice and aroma-bearing vapor. The stripped juice is cooled in *E* and collected for future use. Or if it is to be concentrated immediately, it is flashed direct from *D* into a vacuum feed tank for the evaporation. The vapor from *D* passes to a fractionating column, *F*, equipped with the customary condenser and reflux splitter. A small portion of the reflux is metered off from the splitter, cooled, and collected as product ("essence"). The vent gases from the condenser are cooled, then scrubbed in *G* by countercurrent contact with chilled water, instead of by chilled essence as in previous apparatus. This water is obtained from the bottom of the fractionating column, and after having been used for scrubbing is returned to the column to recover the aroma.

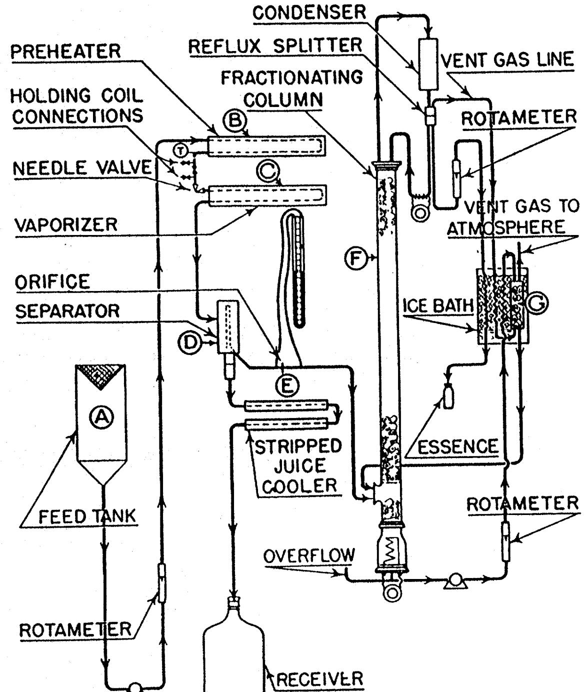

Figure 1. Experimental Unit for Recovery of Volatile Flavors

DETAILS OF APPARATUS

Juice Feed. The juice from which volatile flavor is to be recovered is charged through a conical fine-meshed screen into the stainless steel feed tank, *A*, which is of about 12-gallon capacity. The screen should be of as fine a mesh as the juice will pass through. The cover of the tank is tight-fitting to minimize aroma loss. The juice is delivered to the recovery system from the feed tank by a stainless steel positive delivery adjustable speed pump, the rate of flow being metered through a rotameter calibrated for the particular juice used.

A positive delivery pump is preferred over a centrifugal for two reasons; (1) its delivery is not seriously affected by accidental partial obstruction of the pipelines, and (2) its uniformity of delivery helps to minimize surging in the single tube evaporator. It is very important that juice be fed to the system at a constant rate, as the product (essence) is drawn off at a constant rate and it is the ratio between juice feed rate and product drawoff rate, after steady running conditions have been reached, that determines the strength (fold) of the essence.

Evaporator. The evaporator consists of a preheater and a vaporizer. In earlier work juice heating and vaporization were done, for convenience, in a single tube, but later calculations for the improved design predicted, and experience confirmed, that the time for heating and vaporizing, and hence likelihood of heat damage, are reduced if the juice is first separately heated in a tube of much smaller diameter than the vaporizer tube. Furthermore, uniformity of heating of all parts of the juice stream is achieved by choosing a preheater tube diameter small enough to ensure turbulent rather than streamline flow while the juice is in the harmful range of temperature. This avoids the long heating of the thin layer of juice which, under conditions of streamline flow, travels slowly along the wall of the tube. The tube diameter in this apparatus is about as small as is practical—viz., [illegible]-inch inside diameter. For this tube, calculations based on apple juice of 14° Brix and 2.6 centipoises viscosity at 70° F. (21.1° C.) show that at a flow rate of 10 gallons per hour the juice entering the preheater at that temperature will be in turbulent flow, the Reynolds number being 2080. At a flow of 5 gallons per hour, the Reynolds number near the entrance is only 1040 and consequently streamline flow exists at that point; however, as the juice becomes heated its viscosity decreases and the Reynolds number increases until at the return-bend connecting the two straight portions of the tube it is about 2400 and turbulent flow commences. Thus in the high-temperature portion of the preheater, which is all that matters, local overheating is avoided.

The steam pressure on the jacket of preheater *B* is controlled so as to heat the juice to about 210° F. (99° C.). The hot juice then enters vaporizer *C*, the tube of which is 0.444 inch in inside diameter. Sufficient steam is used in the jacket of *C* to vapor the desired percentage. For each sort of fruit juice, experiments are needed to determine what percentage of vaporization is required to release substantially all the volatiles; for apple juice about 8% is adequate, for some other fruits and berries as much as 20 or 25% is required, and for the heat-developed aroma of processed grape juice special procedures are necessary The vaporization in *C* is measured by metering the rate or flow of vapor from the liquid vapor separator, *D*, by means of an orifice plate and a manometer.

Using separate tubes for the preheating and vaporizing operations in the manner described enables each to be designed closely

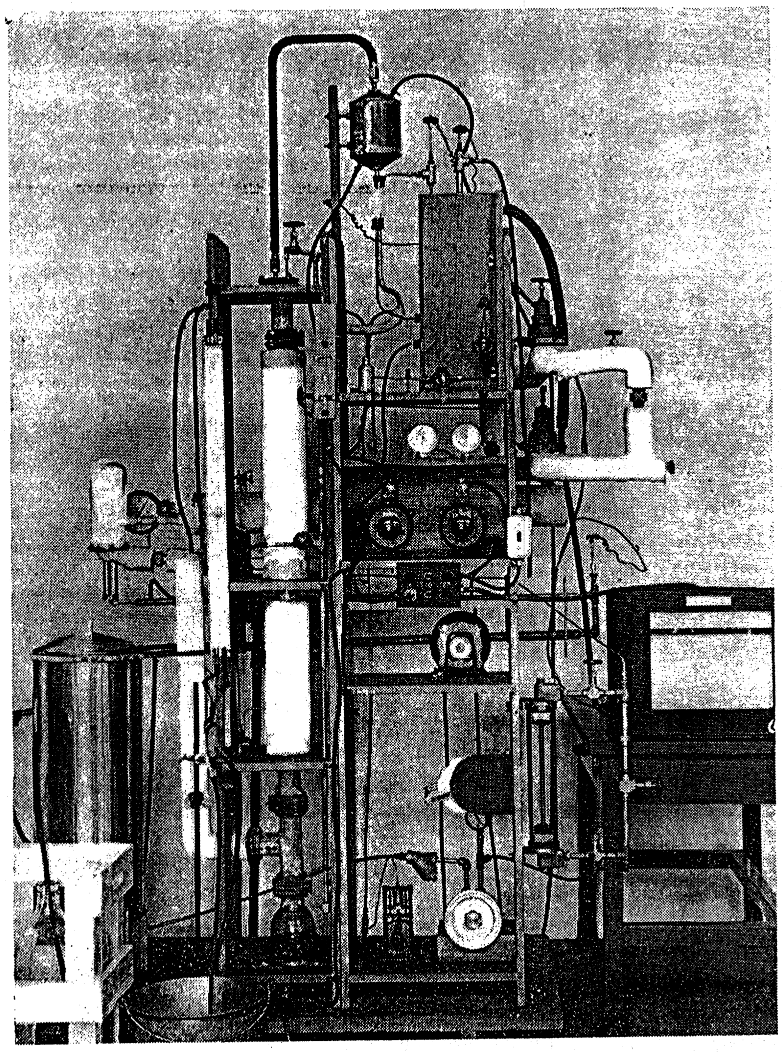

Figure 2. Experimental Unit for Recovery of Volatile Flavors

for its own function, choosing a much smaller diameter for the preheater tube. By this means and by careful design of every part of the juice passages, the authors have been able to reduce the time that the juice remains at high temperature in the apparatus from about 20 seconds to about 2.4 seconds. This obviously reduces greatly the heat treatment that the juice inevitably receives in passing through the apparatus, and consequently minimizes the possibility of damaging its flavor. Many fruit juices develop a cooked taste and aroma on being held at 210° to 215° F. (99° to 102° C.) for 15 to 30 seconds.

Pulsation of the flow through the evaporator, which has given trouble in some essence units which do preheating and evaporating in the same tube, is eliminated by the use of a separate preheater controlled so as to heat the juice nearly to its boiling point, and by having the feed pump of the positive displacement type rather than centrifugal. In a single tube which is fed with cold juice and which is required to heat it and vaporize a portion of it, there is always a tendency to pulsation unless the tube is fed by a pump of absolutely constant flow. Any small variation in flow will be self-amplifying as long as the tube is supplied with juice under constant pressure and discharges its vapor into a constant-pressure chamber. Any increase of flow of cold juice into the tube requires the tube to carry more heating load, and consequently the point of ebullition moves forward in the tube—i.e., moves away from the entrance. The tube surface available for vaporization is thus reduced, and consequently the rate of vapor flow is decreased. This reduces the total friction in the tube, because the friction of the liquid per unit of length is less than that of the same weight of vapor-liquid mixture. This permits a still further increase in juice flow if the pump will supply it, thus amplifying the original small variation. This phenomenon will also occur in a commercial essence recovery unit having several such tubes in parallel, even if the pump is of constant-volume type; the decrease in flow through one tube forces more of the juice supply through other tubes, until the first tube may give nearly complete vaporization of the juice entering it instead of only 10 or 20%. The use of a preheater tube of small diameter and consequently high friction obviously prevents the self-amplifying action described. It adds to the system a friction which increases with increasing flow of juice, thus stabilizing the flow.

Holding Coil. Because there is some indication that volatile flavor may actually be generated in certain fruit juices by prolonging heating, connections are provided between *B* and *C* for a jacketed holding coil which can be of any length required to achieve the desired holding time. This coil also enables the determination of maximum time permitted by heat-damage considerations; this figure is useful in design of commercial plants. In determining whether or not a juice has been damaged by any given time of exposure to any given temperature, it is not sufficient merely to examine the juice immediately after processing. Its behavior on long storage must also be studied to establish whether or not deterioration is accelerated by the heat treatment.

Stripped Juice Cooler. The mixture of liquid and vapor from the vaporizer is separated in *D*, from the bottom of which the stripped juice flows to a two-stage cooler, *E*. In order to cool the juice quickly to a harmless temperature, the tube in the first section of the cooler is of small diameter, 0.152 inch inside. The high velocity here increases the heat-transfer coefficient as well as decreases the contained volume, but causes a high friction drop. Therefore, to avoid the use of a pump at this point, the latter part of the cooler, where temperatures are harmless, is of larger diameter, 0.493 inch inside. In the authors' work this has given satisfactory flow by gravity, but for juices of exceptionally high viscosity a pump might be necessary. If placed between the two sections of the cooler, the possible difficulty of vapor cavitation in the pump will be avoided, provided that the friction drop in the first section is not too high. Cold water is fed through the jackets of the two coolers in series, parallel to the flow of the juice, to increase the rapidity of the initial cooling. The cold stripped juice is received in a stoppered carboy for subsequent processing into a concentrate.

Heat Exposure Time of Juice. In the entire apparatus, the juice is held at or near boiling temperature only while in the vaporizing tube, the separator, the adjacent few inches of the preheater and cooling tubes, and the piping connecting these. At a feed rate of 10 gallons per hour, this time totals about 1.6 seconds, at 5 gallons per hour about 2.9 seconds. One large holdup included in these figures is the film of stripped juice on the walls of the separator below the inlet. This cannot be calculated exactly, because of the disturbing effect of the vapor stream, but an approximate calculation based on gravity flow indicates about 0.6-second holdup at 10 gallons per hour or 1.0 second at 5 gallons per hour.

From a functional viewpoint, in considering the total heat treatment one must also include the heat equivalent of the time spent at lower temperatures while heating and cooling. The flavor changes are believed to be chemical reactions rather than biological processes; on this basis the best assumption is that their reaction rate is halved for every decrease of 10° C. (18° F.). Thus from the time-temperature schedule of the juice in its passage through the preheater and cooler we calculate, for a feed rate of 10 gallons per hour, that the total heat treatment in the preheater is equivalent to that of 0.25 second at the boiling point of the juice (usually about 215° F.), or 0.5 second at 5 gallons per hour. The corresponding figures for the cooler are 0.5 and 0.9 second. Adding these figures to the previously calculated actual holding time at boiling point gives for 10 gallons per hour a total figure of 2.4 seconds, or for 5 gallons per hour 4.3 seconds, representing equivalent time at the boiling point. These figures are not exact, but give a good idea of the rapidity with which the juice passes through the danger zone. This treatment is, however, adequate to sterilize the stripped juice. Bacteriological tests showed complete destruction of yeasts and of all bacteria capable of surviving in the juice.

The stripped juice cooler was, as described, designed for gravity flow. If it ever proves necessary to reduce still further the total heat treatment time, the first section of the cooler could be made of still smaller diameter and a positive rotary pump installed between the two sections. The entire heat treatment due to cooling is eliminated if the stripped juice is immediately to be concentrated, for in that case it will be flashed directly from the separator into a closed tank for feeding to the evaporator, the same vacuum being maintained on the tank as in the evaporator

Flavor Rectifier. The vapors leaving the liquid vapor separator are already partially concentrated with respect to volatile flavor constituents.

For further concentration they enter the fractionating column, *F*, above the stripping section. This is a 2-inch inside diameter borosilicate glass tube, the upper 52 inches of which are packed with $^3/_8$-inch ceramic Raschig rings. For higher fractionation efficiency, $^1/_4$-inch rings may be used, but the safe working capacity of the apparatus will thereby be limited to 9 gallons per hour at 10% vaporization or 4.5 gallons per hour at 20% vaporization. The lower, or stripping, section contains about 6 inches of the same packing. Below the stripping section there is held in the column a pool of liquid in which is an electrical heater of 0.5-kw. capacity, forming a reboiler. This arrangement supplies heat to offset radiation losses from the column and to vaporize the liquid returned from the vent gas scrubber.

Vapors leaving the top of the column are totally condensed and passed to a glass reflux splitter, from this the desired quantity of condensate is drawn off as finished essence, and the remainder is returned as reflux to the column, reheated electrically if desired. For exampl , in making 150-fold essence from apple juice, if the juice feed rate is 10 gallons per hour, and the vaporization 10%, 1 gallon per hour would be vaporized and 0.067 gallon per hour would be drawn off as essence, leaving 0.933 gallon per hour to be returned as reflux. The "reflux ratio" would thus be approximately 14 to 1. The product or essence is drawn off from the reflux splitter through a flowmeter for measurement and control, then passes through an ice-jacketed coil to a receiver which is kept cold

Vent Gas Scrubber. Because the starting juice will contain a certain amount of dissolved air, which must be vented from the system and which will be saturated with volatile aromas at the temperature at which it leaves the apparatus, this air must be cooled and scrubbed before it is discarded. Earlier work (*4*) showed that in the case of apple juice these aromas could be effectively scrubbed out of the air by chilled essence. However, in the case of certain other fruit juices, notably Concord grape and strawberry, this method of scrubbing was not completely effective; the aroma content of the air leaving the system in equilibrium with chilled essence was still appreciable. Therefore, in order to have the vent gases leave the system free from volatile aroma it was necessary to scrub them countercurrently with ice water.

In order to avoid the introduction of extraneous water, which might contain undesirable odors, a part of the column bottoms is utilized. This is pumped through a flowmeter to an ice-chilled scrubbing column, *G*, by a positive delivery pump. A bellows pump is shown in Figure 2, but a rotary type could be used equally well. The scrubbing tower is a glass tube 1 inch in diameter and 12 inches tall, packed with $^1/_4$-inch Berl saddles. The chilled column bottoms enter the top of the scrubber, and the vent gases, previously chilled by passing through a coil immersed in an ice bath enter the bottom and flow up in countercurrent contact with the liquid. Adequate scrubbing of the vent gases is obtained when the quantity of column bottoms used is sufficient to wet th packing thoroughly. The scrubbing liquor from packed tower *G* flows by gravity to the fractionating column and enters it just above the stripping section, in order that the aroma which it contains may be concentrated and recovered as essence.

GENERAL

All parts of the equipment which come in contact with the juice, the vapors, or the essence are made of stainless steel, glass, neoprene, or, in the case of the packing, ceramics. The unit was built in the laboratory shops at a materials cost of approximately $650.

Detailed construction drawings for this unit may be had by writing to the Eastern Regional Research Laboratory, Bureau of Agricultural and Industrial Chemistry, U. S. Department of Agriculture, Philadelphia 18, Pa.

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